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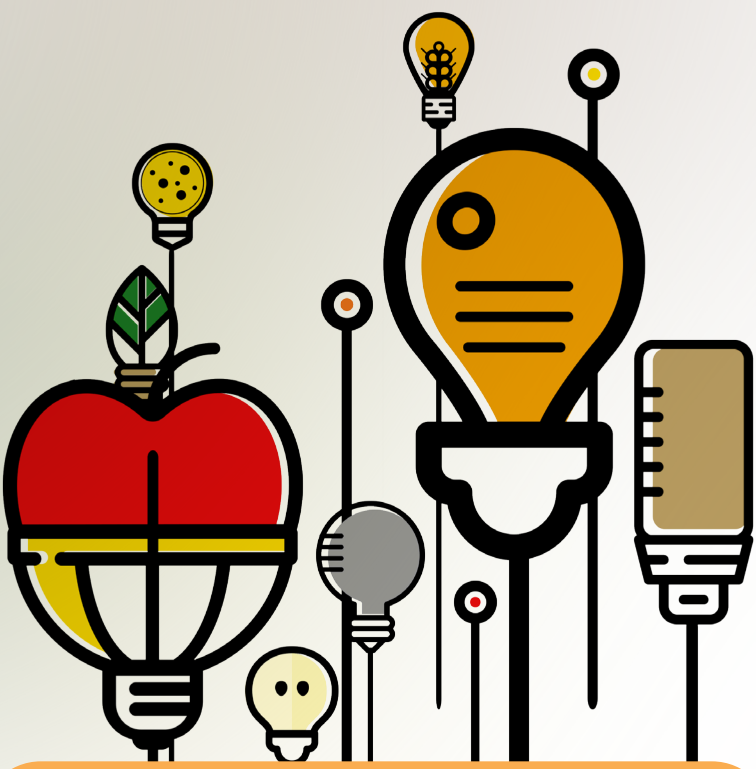

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A COMPARATIVE STUDY ON THE EFFECTS OF BENTONITE AND SUGAR BEET PULP APPLICATION IN MOLASSES PURIFICATION TREATMENT

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ABSTRACT

The reduction of non-sugar compounds content in all intermediate and by-products of the sugar industry, especially molasses, shifts the production towards sugar yield increase and also facilitates betaine recovery. In this respect, the application of possible non-sugar compounds adsorbents is being intensively explored. The presented study investigates the application of sugar beet pulp and sodium bentonite as adsorbents in sugar beet molasses purification treatment. Potential reduction of non-sugar compounds content in molasses was monitored through measurements of molasses colour and turbidity.
The experiments were conducted according to the Box-Behnken experimental design and the effects of independent variables: pH (3−7), adsorbent concentration (3−7 g/L) and molasses dry substance (30−50°Bx) on the sugar beet molasses colour and turbidity were determined by response surface methodology. Applied temperature (60°C) and mixing speed were kept constant throughout the experiments.
The most pronounced impact on molasses colour and turbidity was expressed by pH, regardless of adsorbent used. Low pH treatments improved molasses colour reduction while opposite effect was observed regarding turbidity reduction. More efficient molasses colour reduction was noticed upon sodium bentonite addition compared to the sugar beet pulp. Conversely, greater molasses turbidity reduction was observed upon sugar beet pulp application.
Keywords: *sugar beet molasses, decolourisation, turbidity, bentonite, sugar beet pulp*

INTRODUCTION

Sugar beet molasses represents one of the most valuable by-products of the sugar industry due to the large amounts of fermentable sugars (50−55%) (Šárka et al., 2013). Furthermore, presence of nitrogenous compounds, as well as variety of organic non-sugar compounds with market value also contributes to the molasses value. A strong tendency towards molasses full exploitation through separation of the present valuable compounds has always been a motive for industry and scientific community. The corresponding tendency led to the development of numerous processes in terms of sucrose recovery from molasses likewise betaine and other compounds isolation (Kukić, 1995).
Difficulties in the separation of the desired compound from colourants (alkaline degradation products of hexoses, melanoidins and caramels) present in molasses are usually introduced. In order to achieve the satisfactory purity of the compound of interest, molasses is often subjected to a purification treatment. The aim of purification is to reduce colourants content in molasses and enable easier separation and increase in desirable compound yield. The application of large specific surface area adsorbents for colourants removal from sugar juices was explored in the past decade (Mudoga et al., 2008; Arslanoglu & Tumen, 2012; Jahed et al., 2014). Nevertheless, utilization of the adsorbents potential in molasses purification treatment is a subject scarcely present in the literature and requests further research. In this regard, the present study investigates adsorption potential of the sodium bentonite (Na Bent) and sugar beet pulp (SBP) towards colourants present in sugar beet molasses. The corresponding adsorbents were chosen as readily available low-cost natural materials that could respond to the emerging environmental concerns regarding disposal. Different

bentonite types successfully reduced the content of colour and turbidity causing compounds in sugar juices for more than 25% (Jahed et al., 2014; Erdogan et al., 1996). SBP as lignocellulosic material with high pectin content (24% dry substance) (Tjebbes, 1998) that provides affinity towards cations binding was applied for metal cations and dyes removal (Dronnet et al., 1997; Vučurović et al., 2012). In the treatment of thin juice, as reported by Arslanoglu & Tumen (2012), colour reduction after SBP addition was 7.6% while more successful colour reduction was achieved upon SBP modification with citric acid (23−33%). Treatment conditions also affect efficiency of the applied adsorbent as well as characteristic of the adsorbing compound and could contribute to the adsorption enhancement. In the sugar production process maintenance of adequate pH value is important since decrease in pH leads to sucrose inversion and glucose and fructose formation. As a consequence, besides sucrose loss, the occurrence of Maillard reactions and melanoidins formation in further processing stages at applied high temperatures is enabled (Asadi, 2007).
The aim of the present study was to investigate the adsorption performance of Na Bent and SBP for non-sugar compounds reduction in sugar beet molasses under different treatment conditions. Monitoring of the applied adsorbent and different treatment conditions effects was performed through molasses colour and turbidity measurements.

MATERIAL AND METHODS

Molasses (85°Bx) treated in presented research was sampled from the sugar beet factory “Šajkaška” (Žabalj, Serbia) during the sugar beet campaign in 2016. For the purpose of the experiments, molasses samples dry substance was adjusted to 30, 40 and 50°Bx by using distilled water. As an adsorbent, fine powder formulated sodium bentonite (BP Bentonite-Na, “Bentoproduct” Šipovo, Bosnia and Herzegovina) with montmorillonite content 88–92% and moisture content 9–10% was applied. The bentonite suspensions used in research were prepared according to the producer's recommendations: 100 g of bentonite in 1.5 L of distilled water and hydrated according to the procedure described by Djordjević et al. (2018). SBP used as an adsorbent was obtained during the sugar beet campaign in 2014 from the sugar factory “Crvenka” (Crvenka, Serbia) with initial moisture content of 10–12%. Before application, SBP was hydrated, pressed, dried and milled according to the procedure described for non-modified sugar beet fibres production by Šoronja-Simović et al. (2016).
According to the Box-Behnken experimental design (Table 1), prepared Na Bent suspension or SBP were added to 200 ml of diluted molasses in order to reach the resulting concentrations (3, 5 and 7 g/L). Citric acid (50 g/100 mL) (Lach-Ner s.r.o., Neratovice, Czech Republic) was used for samples pH adjustment (3, 5 and 7 g/L). Molasses treatment was conducted in heated (60°C) water bath (GDE Enzymatic Digester equipped with Multistirrer 6, VelpScientifica®, Ustmate, Italy) with agitation during 30 min. After cooling (25°C), samples were filtered through filter paper (nr. 604 ½, Selecta faltenfilter, Carl Schleicher and Schüll, Dassel, Germany) and the obtained filtrates were used for measurements.
Samples colour was determined according to the method given by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) measuring absorbance at the 420 nm wavelength by spectrophotometer (MA 9522-Spekol 220, Iskra, Horjul, Slovenia) and calculated as follows:

$$Colour\ [IU] = \frac{A \times 1000}{c \times b} \qquad (1)$$

where *C* is the colour in ICUMSA unit (IU), *A* is the absorbance of the solution at 420 nm, *b* is the cuvette length or path length in cm (1 cm cuvette for molasses) and *c* is dry substance content determined by refractometer (modell G, Carl Zeiss, Jena, Germany) expressed in g/cm^3 by using brix-density tables for sugar solutions. Molasses samples turbidity was directly measured using turbidimeter (Turb550IR, WTW, Weilheim, Germany) and given in NTU units. Changes in molasses colour and turbidity were calculated using following equation:

$$Quality\ parameter\ reduction\ (\%) = [(QP_i - QP_f)/QP_i] \times 100 \quad (2)$$

where *QPi* is the initial quality parameter value of diluted molasses and *QPf* is the final quality parameter value of molasses sample after treatment. All measurements were conducted in duplicate prior to every set of experimental runs and in the obtained filtrates.

Table 1. The Box-Behnken design applied in the sugar beet molasses treatment with sodium bentonite and sugar beet pulp.

Independent variables	**Levels**		
	Low (-1)	**Medium (0)**	**High (+1)**
pH	3	5	7
Bentonite suspension concentration (BC)/Sugar beet pulp concentration (SBP), g/L	3	5	7
Molasses dry substance (DS), °Bx	30	40	50

The statistical and graphical interpretation of the results was performed in Design-Expert® software 7.0.0 (trial version, Stat-Ease Inc., USA) by applying a second-order polynomial model. Obtained models and coefficients significance was determined by analysis of variance (ANOVA) comparing p-values ($p<0.05$). Model suitability was evaluated by the adjusted coefficient of determination (adjusted R^2) while lack of fit test was applied for determination of the experimental data reproducibility.

RESULTS AND DISCUSSION

Only figures considering statistically significant effects on modeled response variables are presented. The independent variables effects on molasses colour reduction after treatment with Na Bent and SBP are presented in Figure 1.

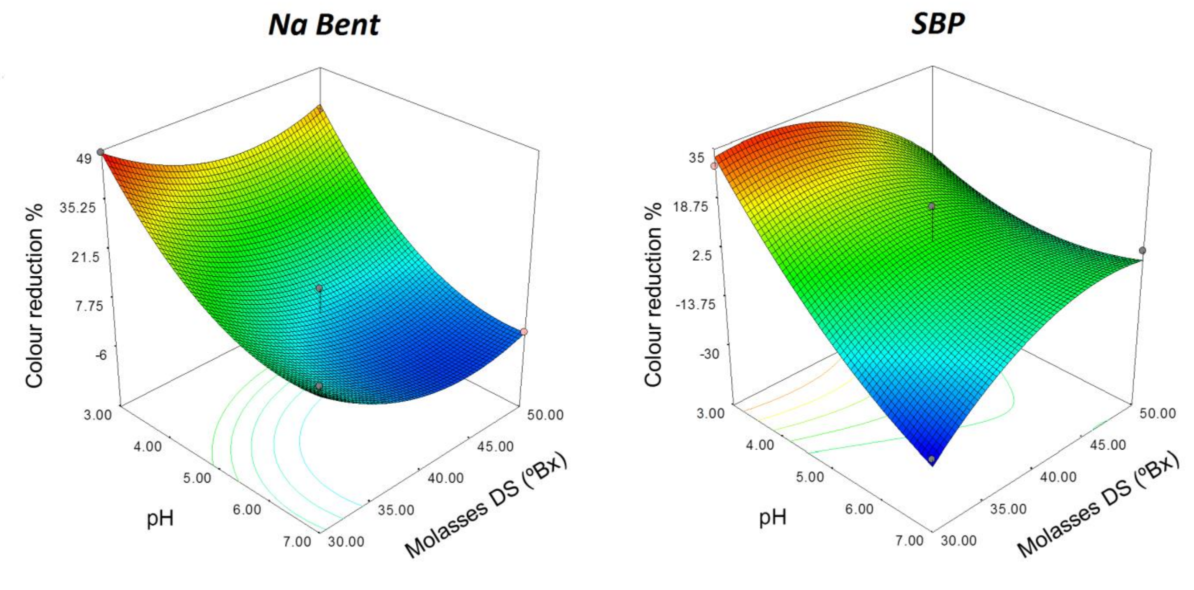

Figure 1. The obtained response surface plots for molasses colour reduction as a function of pH and molasses dry substance

Mainly positive results regarding molasses colour reduction are obtained upon treatment with selected adsorbents at applied conditions. Figure 1 clearly denotes great impact of pH on molasses colour reduction (statistically significant linear effect, Table 2). More successful molasses colour reduction was achieved in treatments conducted at pH 3 compared to other applied pH values, regardless of the used adsorbent (Figure 1). A certain tendency in molasses colour reduction is also noticed at pH 5 although in lesser extent. Successful

molasses colour reduction in the corresponding conditions could be attributed to the electrostatic interaction between positively charged surfaces of the applied adsorbents (Arslanoglu & Tumen, 2012; Lambri et al., 2016) and melanoidines net negative charge in acidic conditions (Migo et al., 1997). Furthermore, the decrease of melanoidines solubility in the acidic environment (Chandra et al., 2008) coupled with Na Bent and SBP relatively high swelling capacities (Lambri et al., 2016; Šoronja-Simović et al., 2016) could also contribute to the melanoidines and other colourants retention. With the increase in molasses DS, tendency towards decrease in colour reduction was observed regardless of the applied adsorbent (Figure 1) (statistically significant linear effect for Na Bent, Table 2). Lower colour reduction was achieved probably due to the occupancy of adsorbent active sites with the increasing colourants content (increased molasses DS). The increase in number of adsorption sites can be afforded by the application of higher adsorbent concentration and hence contribute to the better efficiency in colour reduction. Among the applied adsorbents, slightly higher molasses colour reduction was noticed upon the Na Bent application.
The obtained quadratic polynomial models regarding colour reduction were significant at $p<0.05$ (Table 2). Obtained non-significant lack of fit for all conducted experiments ($p>0.05$, Table 2) indicated that the quadratic polynomial models were adequate for predicting experimental data reproducibility. Suggested models suitability was also confirmed by relatively high values obtained for adjusted R^2 (Table 2).

Table 2. Regression equation coefficients of the second order polynomial model for responses (actual factors) and analysis of variance (ANOVA) of the independent variables effects on modelled response variables obtained after sugar beet molasses treatment with sodium bentonite and sugar beet pulp.

	Response			
	Colour reduction [%]		**Turbidity reduction [%]**	
Coefficient	**Na Bent**	**SBP**	**Na Bent**	**SBP**
Intercept				
β_0	252.56	95.87	-225.38	-27.56
Linear				
β_1	-37.90*	-58.52*	35.71	106.60*
β_2	6.12	-13.55	28.14	43.64
β_3	-6.67*	5.33	7.34*	-16.22
Quadratic				
β_{11}	2.84*	1.79	3.24	-13.49*
β_{22}	-0.10	1.14	/	-4.97
β_{33}	0.09*	-0.11	/	0.04
Interaction				
β_{12}	0.61	0.76	1.36	-0.65
β_{13}	-0.07	0.71*	-1.65*	1.72
β_{23}	-0.20	/	-0.89	0.31
Model				
p-value	0.0124*	0.0174*	0.0126*	0.0215*
df Model	9	8	7	9
Lack of fit	0.9326	0.9834	0.1111	0.5695
df Lack of fit	3	4	5	3
Adjusted R^2	0.8932	0.8115	0.7789	0.8574

Na Bent-sodium bentonite; SBP-sugar beet pulp; df-degree of freedom
**Values are significant at p<0.05*

The effects of independent variables and their interactions regarding molasses turbidity reduction upon Na Bent and SBP addition are presented in Figure 2. The influence of pH on molasses turbidity reduction was also pronounced especially upon SBP application (statistically significant linear and quadratic effects, Table 2).

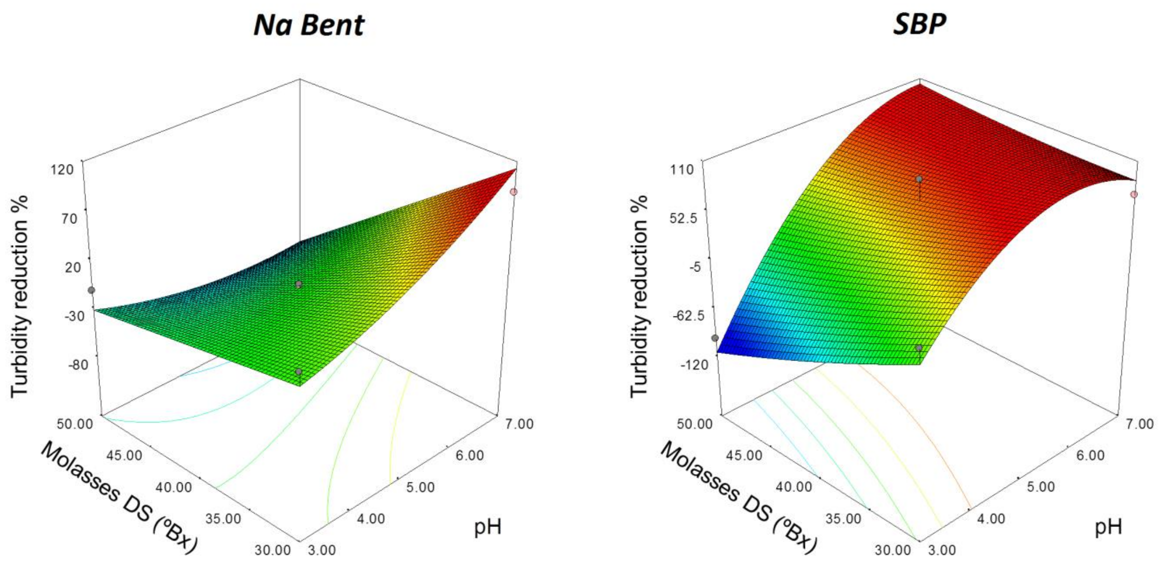

Figure 2. The obtained response surface plots for molasses turbidity reduction as a function of pH and molasses dry substance

Conversely to colour reduction, with the increase in pH more successful molasses turbidity reduction was observed, regardless of the applied adsorbent (Figure 2). Maximal molasses turbidity reduction was reached when treatments were conducted at pH 7 (~89%) regardless of other examined conditions. At pH 5 tendency towards turbidity reduction was also noticed especially after SBP application. The diversity of turbidity causing compounds present in molasses and their characteristics at certain pH affect the adsorbents performance. Furthermore, the prevailing mechanism for turbidity causing particles reduction is primarily associated with the retention of turbidity causing compounds inside the adsorbents porous structure. SBP was established as more efficient compared to sodium bentonite regarding turbidity reduction probably due to the highly porous structure and presence of multiple functional groups. Impact of molasses DS on turbidity reduction was less pronounced (statistically significant linear effect only for Na Bent, Table 2). Nevertheless, achieved turbidity reduction was greater in molasses samples with minimal DS regardless of the applied adsorbent (Figure 2). The impact of applied adsorbent concentrations on turbidity reduction was smaller compared to the impact of DS and especially pH.
The obtained quadratic polynomial models regarding turbidity reduction were significant at $p<0.05$ (Table 2). Obtained non-significant lack of fit ($p>0.05$, Table 2) indicated that the quadratic polynomial models were adequate for predicting experimental data reproducibility. Relatively high values obtained for adjusted R^2 (Table 2) confirm the suggested models suitability.

CONCLUSIONS

The presented study demonstrated positive outcome of the sodium bentonite and sugar beet pulp application in the reduction of non-sugar compounds from sugar beet molasses. The degree of molasses colour and turbidity reduction was primarily affected by the applied pH during treatment and dry substance content of the treated molasses. Conversely to turbidity reduction, more efficient molasses colour reduction was obtained in strongly acidic conditions regardless of the applied adsorbent. In order to achieve balance between the satisfactory molasses colour and turbidity reduction after purification, further investigations regarding favourable treatment conditions have to be undertaken.

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